Energy, Mines and Resources Canada

Hon. Marcel Masse, Minister

Énergie, Mines et Ressources Canada

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Canada

CANADA'S THERMOCHEMICAL CONVERSION R&D APPROACH

R.D. HAYES
Bioenergy Research and Development
Renewable Energy Branch
Energy, Mines and Resources Canada
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ABSTRACT

Canada's biomass thermochemical conversion R&D is funded primarily through Energy, Mines and Resources Bioenergy Development Program. The thermochemical area has undergone considerable transformation over the past decade especially with respect to changes in expectations from long-term potential to mid-term application. Main stream thermochemical projects are reviewed, as are associated combined thermochemical/biochemical projects. These are described also in the context of overall Canadian bioenergy R&D. In order to improve the analysis of cross-disciplinary feedstock/ process/product combinations, a novel methodology for strategic planning of R&D is being implemented. These combinations are mapped out in what are called Prospective Applications in Technology or PATs. Selected PATs can be analysed more rigorously as to technical expectations, current and future economic and market potential, and estimates of likely timeframes for commercialization can be developed for various scenarios.

INTRODUCTION

As we uncover more and more about what can be done with biomass and wastes that cannot be done with coal, strategic planning of technical priorities and activities in research, development, and demonstration is becoming increasing complex. Gone are the days when planners could organize their thoughts by a few simple and distinct technology descriptors limited to air and oxygen gasification, pyrolysis, or acid depolymerization followed by yeast fermentation.

In the thermochemical area of ten years ago, support was provided to technologies that showed promise in substituting merely for natural gas or crude heating oil. With biomass thermochemical conversion development in its infancy, the theoretical promise of higher valued specialties (e.g. fine chemicals) and commodities (e.g. high octane gasoline) appeared to be well off in the future in terms of economic reality.

Within the past decade the science has become better understood and technical advances in thermochemical process development have accelerated beyond earlier anticipation. A new world of conversion and product opportunities has begun to reshape our view of the timeframe for commercialization. From a very long term perspective as recently as five years ago, we are beginning to see a few selected commercial prospects emerge (e.g. pyrolysis of used tires). Perhaps yet to demonstrate the greatest impact on biomass and wastes conversion is the separate processing of fractionated lignocellulosic derived materials such as cellulose, xylans, lignins and extractives.

Scientists and engineers have broadened the scope of experimental conditions and gained much deeper understanding of chemical transformations of the complex feedstocks and multiple products from biomass and waste conversion. More effort is also being placed in alleviating waste disposal problems (and costs) by transforming them into sources of net revenue. By no means excluded from this effort are the waste effluents produced by biomass conversion processes.

Researchers, industry and government have all become more creative in building a diversified yet comprehensive portfolio of thermochemical conversion opportunities for the commercial world. Though nothing thoroughly new has emerged, the past ten years have seen an evolution from a subject of technical curiosity to one of concerted effort toward cost effective products for the market place.

PROSPECTIVE APPLICATIONS OF TECHNOLOGY (PAT)

A broader diversified portfolio as referred to above presents a major challenge to the R&D planner. Added to this challenge is the ever present decrease in availability of R&D funds. These two challenges have necessitated a major overhaul in organizing R&D priorities in the context of assessing both near and long term commercialization opportunities and needs.

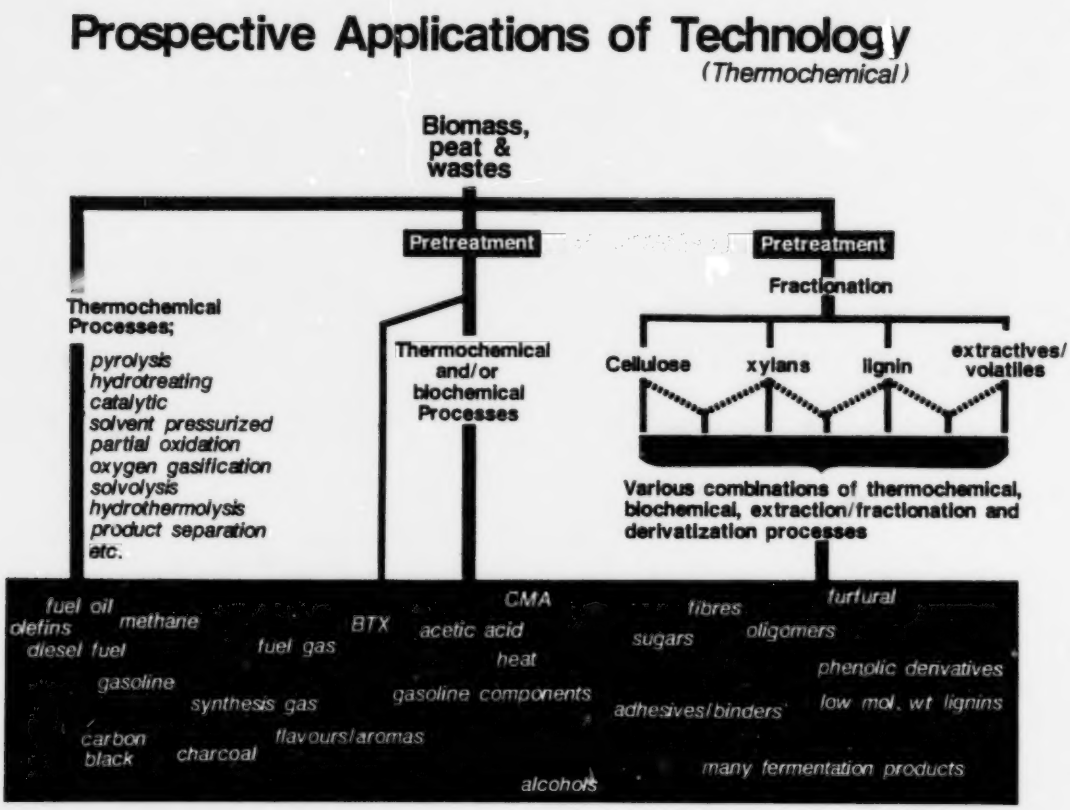
The commercialization process for a new technology as defined within the Bioenergy Division of Energy, Mines and Resources Canada includes everything germaine including opportunity identification, the actual research, development, technical evaluation, and demonstration as well as the determination of current and projected economic feasibilities, current and future market, expected timeframes for commercialization, and other factors affecting market introduction and penetration. Once these elements have been assessed and analysed for the various feedstock/ conversion technology pathways, priorities are to be established with respect to activities that government can, should, or must, do and those that government should not, or cannot afford, to support.

The first step in the process is to organize the three basic elements of a technology by resource (eg. biomass, wastes, peat etc.), process(es), and product(s) for market application. The particular feedstock combined with specific conversion processes and a particular market application result in what we define as a **Prospective Application of Technology or PAT.** When the various PATs are viewed together, the

picture can become rather confusing. This is especially so when considering non-traditional thermochemical opportunities or combinations with biochemical technologies that might be otherwise overlooked during conventional analysis. In order to simplify the process we can illustrate many of the various combinations pictorially in what we call a **'PAT map.'**

Referring to Figure 1, which is a simplified version of a **'PAT map'** for thermochemical technologies, one can visualize perhaps one hundred or more probable combinations of resources, processes and product applications. The product possibilities alone include a vast array of oils, gases, chars, carbon black, sugars, fermentation products, high value specialty chemicals, medium value commodity chemicals, olefins, gasoline and gasoline components, diesel fuel, and materials.

FIGURE 1



Our view of the thermochemical world is no longer limited to merely the pyrolysis or gasification of wood to oil or gas. Thermochemcial conversion is not treated in isolation from biochemical, fractionation or derivatization processes. Even considering a single process technology, the variation in process conditions, catalysts, reactants, or feedstocks can impact the range and output of possible product combinations.

The second step is to select a handful of promising PATs, and perform an indepth analysis of expected technical progress, economic feasibility, market penetration, and timeframe for commercialization. In theory, this should provide government (and industry) with a rationale for desirable government (and industry) activities in those such as research, development, demonstration, detailed engineering, market studies, economic and financial analysis, information dissemination, or other incentives.

Some of the so-called 'thermochemical PAT's' (partial or full) that are not typically represented at thermochemical conversion of biomass conferences, but which are included or planned for in Energy, Mines and Resources' BDP include the following:

- hydrotreating of plant oils (oilseed, tall oil) to produce high cetane diesel rather than a transesterified imitation.
- biochemical and thermochemical derived lignin that is thermally or thermochemically treated with or without catalysts (eg. gasoline, chemicals).
- product derivation of thermochemical and biochemical lignins (eg. resins, adhesives, cement dispersants).
- thermochemical treatment of the steam fractionated cellulose components of biomass.
- direct steam (thermal) production of adhesives with a fibre byproduct suitable for molding, pelletizing, or thermochemical or biochemical processing.
- calcium magnesium acetate (CMA) production from aqueous effluents (ie. dilute acetic acid) of pyrolysis and biochemical processes.
- investigation of wheat chaff as a thermo- or biochemical conversion feedstock.
- methanation of pyrolysis aqueous effluents.
- peat processing into fuel oil and activated carbon.
- separation of short and long fibres of hardwoods where pulp is processed from the long fibres and fuels processed biochemically or thermochemically from the lower value short fibres.
- source separated and/or prescreened, or raw, municipal wastes (including sewage sludge) that undergo thermochemical or biochemical conversion with or without pretreatment/fractionation.

The above listed examples illustrate how difficult it has become to present BDP R&D strategies in terms of distinct line items that we have used in the past such as combustion, fuels processing, thermochemical conversion, and biochemical conversion. Nevertheless government **budgetary planning** requires such a breakdown until a better organizational structure is created.

BIOENERGY DEVELOPMENT PROGRAM ORGANIZATION 1987/88

This section describes the organization of efforts according to funds that were available in 1987/88. Line items are described below according to **budgetary planning** as opposed to the more complicated technical strategies involved in **PAT planning** described earlier since the latter process is only now getting underway.

Tables 1 and 2 show visually and quantitatively where so-called thermochemical R&D dollars were spent in the context of the overall program. Table 3 indicates a rough breakdown of effort in conversion areas according to peat, wood, wastes, and agricultural resources. Overall cost-share with industry, as of November 1987 is outlined in Table 4. The dollars indicated in Table 4 represent total value of current (ie. as of November 1987) R&D contracts, rather than a single year breakdown for 1987/88.

TABLE 1

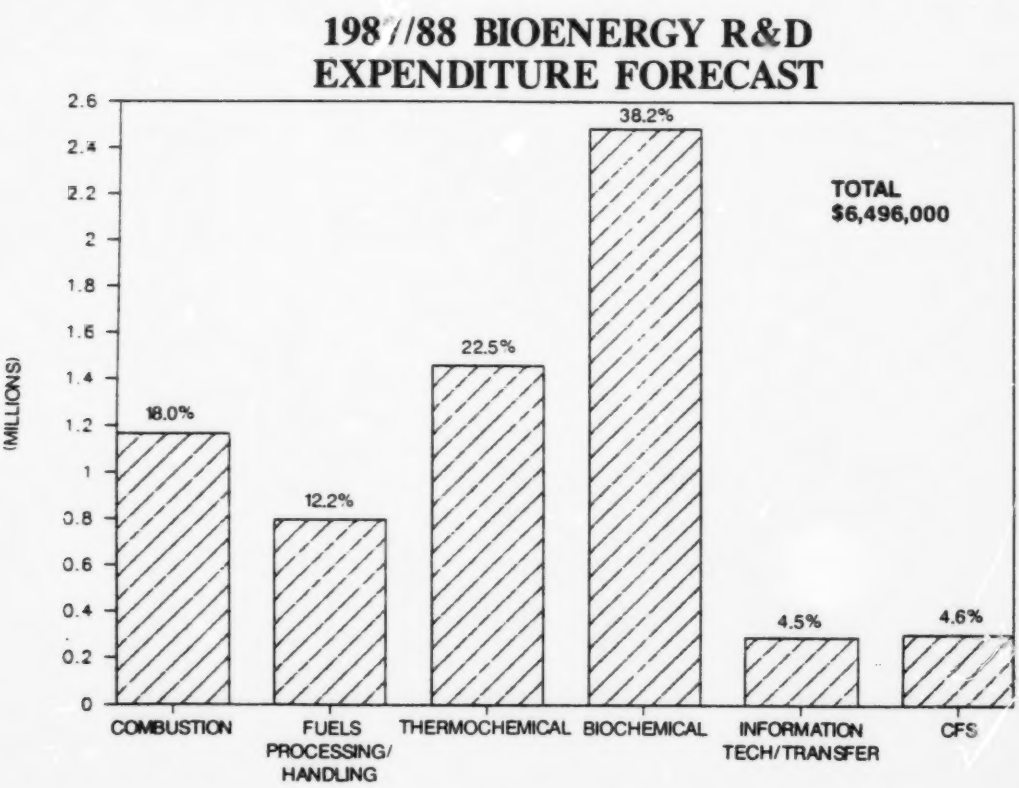


TABLE 2

87/88 Bioenergy R&D *Expenditure Forecast*

	$
Combustion	
Direct Combustion	*690,000*
Residential	*376,000*
NITEP	*101,000*
Fuels processing	
Handling/Processing	*644,000*
PEAT	*150,000*
Thermochemical	
Liquefaction	*762,000*
Gasification	*482,000*
Chemicals	*217,000*
Biochemical	
Alcohols	*2,255,000*
Anaerobic Digestion	*228,000*
Information & Tech Transfer	
I.T.T.	*291,000*
Biomass Production	
To CFS	*300,000*
TOTAL FORECAST	**$6,496,000**

TABLE 3

1987/88 R&D Allocations

By Resource/Technology

Combustion		
Wood	$737,000	63.1%
MSW & Other wastes	$213,000	18.3
Peat	$33,000	2.8
Agriculture	$184,000	15.8
TOTAL	$1,167,000	100%
Fuels Processing/Handling		
Wood	$521,000	65.6%
MSW & other wastes	$0	0
Peat	$187,000	23.7
Agriculture	$86,000	10.7
TOTAL	$794,000	100%
Thermochemical		
Wood	$1,080,000	73.9%
MSW & Other wastes	$153,000	10.5
Peat	$71,000	4.9
Agriculture	$157,000	10.7
TOTAL	$1,461,000	100%
Biochemical		
Wood	$1,872,000	75.4%
MSW & Other Wastes	$209,000	8.4
Peat	$0	0
Agriculture	$402,000	16.2
TOTAL	$2,483,000	100 %

TABLE 4

Overall Cost-Share Estimate (Nov.1987)

($000)

	EMR Share	Contractor share
Combustion	*1450*	*280*
Biochemical conversion	*3550*	*1630*
Thermochemical conversion	*1710*	*210*
Fuels proc. (+peat)	*1270*	*300*

BRIEF REVIEW OF OTHER BDP R&D

Referring to any of Tables 1, 2, or 3, the following is a cursory view of activities in each technical area.

The **combustion** area's main focus of effort is the evaluation and improvement of industrial and residential combustor performance and emissions. This includes work on existing installations as well as new developments such as a rotary sub-stoichiometric two-stage kiln combustion and a boiler design based on the utilization of flax shives as an energy source for drying alfalfa. Also included has been support to Environment Canada's National Incineration Test and Evaluation Program (NITEP) and the development of standards for performance, safety and emissions of residential wood burning appliances.

The **fuels processing** area includes research in biomass materials handling, storage, drying, beneficiation, and preparation. This research is seen to provide necessary infrastructural support for all biomass conversion processes. Next to the combustion R&D, developments in this area have resulted in rapid commercialization by industry in bioenergy (e.g. boiler heat recovery) as well as non-bioenergy applications in the pulp and paper and lumber drying industries. Examples include a bin, silo and non-consolidating feeder system that regulates the controlled and non-disrupted feeding of hog fuel and chips to burners, a device to exclude frozen lumps of hog fuel and chips to burners, a continuous moisture sensor to provide improved combustion control of biomass of varying water content, and a low energy grinder to powder wood suitable for suspension-fired boilers.

The **biochemical** area receives the largest allocation of funds, in part due to increasing industrial leverage provided by cost-shared R&D. Much of the impetus for this area comes from the near to mid-term (1990's) competitive potential to enter the transportation fuel market with biomass derived ethanol blends and neat ethanol fuel along with value added byproducts. The program is divided into a number of areas including enzymatic and acid hydrolysis of lignocellulosics, steam and extrusion pretreatment and biomass fractionation, novel fermentation engineering, biotechnology of enzymes and microorganisms, anaerobic digestion, characterization and derivatization of lignin, and the development of non-ethanol byproduct streams, among others. The rate of achievements in this area over the past four years rivals that in the thermochemical area. The lure and promise of biotechnology and the recent accelerated progress are likely the major factors in attracting industry participation in an area that was viewed by our program as very long term as of only five or six years ago.

CANADA'S MAINSTREAM BIOMASS THERMOCHEMICAL CONVERSION R&D

An approach has evolved in Canada whereby, unlike earlier efforts, a whole range of products and reactants are now considered. Products include gases and oils of varying quality, sugar solutions in high yield, chemicals (olefins, phenolics, as well as high value specialty chemicals), gasoline or diesel fuel, and higher value carbon products. Diversification of reactants include whole biomass, fractionated biomass components, peat, and municipal solid waste, including source-separated wastes such as used tires. Another area of research is the treatment, and especially, the conversion, of waste aqueous effluents from thermochemical conversions into value-added co-products. Left unprocessed, these effluents would otherwise incur a cost for waste treatment.

Gasification

Canadian developments in gasification of biomass for the production of low, medium and high energy gases have enjoyed world technical leadership for the past few years, but there has been a disappointing uptake by Canadian industry. The cautious attitude of our industry appears to have been due largely to premature and unsuccessful commercialization attempts of immature technologies in the U.S. during the latter '70s and early

'80s. A possible revival of gasification technology applications in Canada appears to be for replacing industrial boilers that use oil, for municipal energy from waste projects to provide environmentally acceptable space heating for industrial and municipal complexes, and for applications in remote communities. The work to complete ongoing projects will continue, although at a reduced level, and any impetus for an expanded program will likely wait for stronger industry confidence, interest, and cost share. Application of Canadian technology in developing countries appears to hold some promise.

Large scale gasification technology, developed through the Biosyn project at St. Juste-de-Bretenières, offers a world leading technology in pressurized, air or oxygen fed, fluidized-bed gasification that can be adapted to the production of synthesis gas for methanol production or low energy gas for other applications such as space heating, gas turbines or industrial diesel engine operation.

Another portion of the BDP gasification element is directed to the development of a small scale gasifier that should be capable of using wet wood and producing a clean gas. The Heuristic gasifier is a two-stage reactor that, in principle, operates similarly to a downdraft design. The first stage is an updraft gasifier where producer gas and condensible vapours (tars) are routed through a hot char bed below. The tars are to be cracked in the second stage. The project now underway is to modify the gasifier, optimize the operating parameters, and enhance cracking in the second stage.

Liquefaction/Pyrolysis and Other Thermochemicals

Research in the direct liquefaction of biomass was to wind down as of 1985 because the rapid commercialization that was expected to occur in the early '80s did not materialize. The plan was to conclude as much of the work as possible, revert to a lower cost basic research, and pursue the chemicals from liquefaction option. Then in 1986 there appeared to be a turn around in commercial interest. Unprecedented technical achievements started to renew aspirations of exploiting selected market niches for pyrolysis products. The following is a brief review of current projects.

McGill University

Over the past several years researchers W.J.M. Douglas and D.G. Cooper at McGill University have been studying an interesting thermochemical approach to wood liquefaction using aqueous hydrogen iodide at fairly mild conditions of pressure and temperature (125°C). Still far from certain is the exact nature of the liquid products and the techno-economic practicality of hydrogen iodide recovery and recycle. On the positive side, in addition to the low severity conditions of reaction, the process removes about 80% of the oxygen in the wood, and the char yield is very low. Without industry involvement, this project is to be supported at a minimum level for the next two years to enhance understanding of the reactions and improve process conditions.

University of Toronto

D.G.B. Boocock and co-workers at the University of Toronto have undertaken the investigation of a steam pyrolysis or hydrothermolysis of wood. Based on their earlier work, they recently designed and constructed a laboratory scale cascade autoclave which can accommodate up to 100 g of wood chips or 170g of a single larger piece (3.8 cm reactor I.D., 600ml volume). It is rated at 24.1 MPa (3500 psi.) at 350°C allowing for 7.6 MPa (1100 psi) gas overpressure above the vapour pressure of water at that temperature. Results to date are preliminary since the unit was commissioned only in 1987. Replicated results indicate that oil yield increases with increased chip size. Dry product oil yields are high (up to 50%) with no solids contamination, and the oil is easily separable from the aqueous phase. Coupled with an upgrading process, this technology may someday lend itself well to commercialization.

In addition to their process development work, Professor Boocock's group has contributed greatly to the basic understanding of biomass liquefaction, especially through their scanning electron microscopic studies. The group at University of Toronto is also performing upgrading studies through the catalytic hydrotreatment of model compounds. Of potential interest too is their discovery in 1984 that a particular clone of hybrid poplar yielded 6% phenol. This discovery raises the prospect of matching processes more closely with specific feedstocks in the interest of optimizing a particular product slate. Efforts are now beginning to genetically engineer trees that are designed for specific process/product applications.

Université de Sherbrooke

E. Chornet, R.P. Overend and co-workers at the Université de Sherbrooke have been working on a liquefaction process in pressurized solvent for some years. Their approach involves an overall integration of biomass pretreatment, fractionation, acid processing, thermochemical and biochemical treatment.

Funding for research at Sherbrooke in the areas of peat and wood conversion is provided by a number of sources, including BDP. The program there involves a comprehensive variety of fundamental studies, product engineering and technology development at laboratory and pilot plant levels. Examples of approaches under investigation include a thermo-mechanical-chemical treatment to fractionate and liquefy biomass including steam treatment, thermocatalytic conversion of fractionated products such as lignin to monomers, ethylene glycol solvolysis/liquefaction, acid/thermal/shear treatment of biomass and cellulose, and a variety of biochemical investigations in combination with thermochemical techniques. In association with other laboratories, the chemical engineering laboratory of Professor Chornet is especially well equipped for analytical characterization of products derived from their various reaction systems.

University of Waterloo

D.S. Scott, J. Piskorz, D. Radlein and co-workers at the University of Waterloo are well known for their fluidized bed flash pyrolysis development, also known as the WFPP (Waterloo Fast Pyrolysis Process). A number of companies and the European Economic Community have indicated their interest in collaborating with the Waterloo group. The WFPP actually includes five process options as follows:

1. Direct thermal processing at 450-550°C, atmospheric pressure and about 500 ms vapour residence time. They report the highest liquid yields (80% including water, based on input wood) that is a suitable fuel for conventional boilers.

2. By varying the process conditions and adopting a mild sulphuric acid pretreatment of wood followed by fluidized bed thermopyrolysis, the WFPP produces a high yield and concentration of anhydro-sugars rather than oil products. Their reproducible yields of sugars from pure cellulose are about 80% of theorectical in a concentrated form. One can easily speculate whether this development could challenge some of the equally exciting bioconversion methods of converting lignocellulosics to fermentable sugars. High yields of levoglucosan from cellulose are quite well known, but not from wood. The recent results at Waterloo open many possibilities of cost effective levoglucosan production.

3. Waterloo's hydrogasification work has been technically highly successful, resulting in 75% conversion of carbon from wood to methane via pyrolysis over a nickel-alumina catalyst with hydrogen at about 550°C and 440 ms residence time.

4. Under current investigation is a fourth process option of producing unsaturated hydrocarbons in a catalysed reaction. Apart from the use of catalysts, the process equipment and operating conditions are very similar for all of the above process options.

5. The Waterloo group is also performing upgrading research by catalyticly hydrotreating pyrolysis oil fractions, including lignin, in a continuous pressurized reactor.

University of Western Ontario

Although not currently directly funded by BDP, M. Bergougnou, R. Graham and co-workers have developed an Ultra-Rapid Pyrolysis or Ultrapyrolysis process at the University of Western Ontario. Although there are similarities in this work and the research at the University of Waterloo, there are important differences. Whereas the Waterloo process utilizes fluidized-bed heat transfer, Professor Bergougnou employees a very rapid (30ms) mixing and heat transfer in a vortical contactor or vortactor followed by a plug-flow entrained-bed down flow reactor (50-900ms) and quenching (30ms) with cryogenic nitrogen in a cryovortactor. Also dissimilar to the Waterloo process are the process conditions (650-1000°C, 50-900 ms residence time), and the main product at these temperatures is gas rather than liquid. Since pyrolytic fuel gas production has not been of high priority in Canada's bioenergy R&D strategy, the current objective, in collaboration with Ensyn Engineering, (see below) is production of chemicals and, in particular olefins.

It is interesting to note here that the Universities of Waterloo and Western Ontario conducted an extensive data comparison from each of their reactor systems. Using selected data from both groups at around 500 ms residence time, liquid and gas production data were plotted vs temperature. The temperature ranges were as follows: Waterloo at 400°-750°C, and Western Ontario at 650°-900°C. With combined data for each of the gas yield vs temperature and liquid yield vs temperature respectively there was considerable agreement of data in overlapping regions. In fact a simple first order kinetic model is able to describe the oil yield over the temperature range of both experiments.

Ensyn Engineering

Ensyn Engineering is a recently formed company whose principal investigator, R. Graham has scaled up the University of Western Ontario Ultrapyrolysis reactor by a factor of 20 to a 5-10 kg/hr capacity RTP (Rapid Thermal Processor). The reactor is designed to accept any carbonaceous feed (solid, liquid, or gas) by injecting it into a turbulent cloud of hot solids. The mixed feed plus solids is carried through a tubular transport reactor to an inertial separator where vapour products are removed. This project is also co-sponsored also by the private sector.

Work planned for 1988 involves thermal cracking and catalytic treatment of pyrolysis oils and various lignin preparations including steam exploded biomass derived lignin.

Laval University

Multi-stage vacuum pyrolysis was developed by C. Roy and co-workers, initially at the Université de Sherbrooke and, currently, at the Université Laval. The technology is based on a multiple hearth vacuum pyrolysis process development unit located near the Université Laval and operating at a 30 kg/hr biomass capacity. Some additional R&D support is currently provided by two Canadian companies. There is also an industrial cost-shared pilot demonstration of a single-stage process at St-Amable, Quebec. The unit has a capacity of 200 kg/hr and is designed for used tires.

Although the multiple hearth concept suffers from low heat transfer relative to other pyrolysis processes, and, at first glance, is capital cost intensive, it has a number of redeeming features that show commercial promise as follows:

1. A high yield of pyrolysis oil (50% based on wood).

2. The production of co-product carboxylic acids and high value chemicals.

3. Reactive charcoal at 25% of input wood.

4. The aqueous phase is recovered separately as vapour or liquid as an integral part of the process leaving a near water-free pyrolysis oil ready for upgrading.

5. The multiple hearth performs a product fractionation function that could reduce extraction costs of high value chemicals.

Centralized Analysis

One final work of interest in Canada is the Centralized Analysis project at B.C. Research. In 1984, a trial project was set-up whereby different Canadian bio-oils could be compared in a standard manner. The project embraced a three-pronged approach. Under the guidance of J. Howard and J. McKinley, B.C. Research performed and/or coordinated the centralized analyses of optimized oils produced by each researcher. If a particular analytical capability was not available at B.C. Research, another laboratory with that strength was sub-contracted to do it. Individual researchers also did some of their own analyses to obtain immediate experimental feedback.

The second prong of the approach was that all researchers were provided with a standard wood sample Populus deltoides by Forintek Canada Corp. The idea was that when each process development became somewhat optimized, the researcher would submit oil from the standard wood sample to the centralized analysis team.

The third prong was a computer communications network link called CoSy, through the University of Guelph, to provide fast communication of analytical data. It was also used to encourage multilateral and bilateral collaboration and problem solving.

The centralized analysis project is entering Phase II. Learning from the successes and pitfalls of the first two and one half years, the scope of this project has changed somewhat. The consensus of program managers and researchers is that Phase II will have two major tasks.

Task 1 is a set of analytical techniques along the same lines as the initial project except the methods that were considered to be less interesting to the entire group are not included. The basic analyses of task 1 include the following:

- Elemental Analysis
- Water Content
- Density
- Carboxylic Acids
- Gas Liquid Chromatography
- Carbon-13 NMR
- Gel Permeation Chromatography

Task 2 consists largely of special analytical projects to meet the special needs of individual researchers, the Bioenergy Development Program or groups of thermochemical conversion researchers that may arise over the next two years.

INTERNATIONAL VIEW - IEA

Under the International Energy Agency, eleven countries signed a three year Bioenergy Implementing Agreement, effective January 1, 1986 to December 31, 1988. Canada, USA, Sweden and Finland agreed to collaborate on a project entitled Direct Biomass Liquefaction (DBL). A Working Group of engineers and other specialists are preparing a detailed technical-economic assessment (TEA) at commercial size scale-up and operation of the most promising high and low pressure pyrolysis processes. Both primary oil production and upgrading are considered in the TEA. The upgrading work on Canadian atmospheric and vacuum pyrolysis oils has been conducted by D. Elliot at Battelle Pacific Northwest Laboratories. Yields of products in the gasoline boiling range have so far reached 35% of primary oil by hydrotreating. The Working Group is attempting two types of analyses, one based on current state of the art and the second based on projected improvements and developments in the technologies.

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REFERENCES

This paper contains technical contributions including progress reports from all the Canadian and other workers mentioned here. The reader is referred to these individuals or their published papers as primary references.